

Istituto Nazionale di Fisica Nucleare

Laboratori Nazionali del Gran Sasso

DarkSide Project Process Procedure

**LSV Cleaning, Drying, Filling,
Loop Purification, and Draining**

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Procedure Author(s):

Marco Carlini (Maintenance Manager)

Reviewed by:

Cristiano Galbiati (Spokesperson)

Peter Meyers (Operations Leader)

Federico Gabriele (GLIMOS/RAE)

Last Revised and Approved by:

Augusto Goretti (Operations Manager)

Procedure validity:

from Revision Date
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2.Revision History

Revision #	Date	Author(s)	Rationale	Sections Updated
1	Mar, 6 2015	CK, AG, FG	First draft document	All
2	Mar, 9 2015	FG	General revision	Section 8
3	Mar, 16 2015	FG	General revision	All
4	Mar, 17 2015	MC	General revision	All
5	Mar, 19 2015	MC, PM	General revision	All
6	Mar, 22 2015	MC, PM, CG	General revision	All

3.Description

This procedure describes the operations of the removal of the Atmospheric Argon (AAr) in the DS-50 detector, the purge of the DS-50 detector (and other connected volumes), and the DS-50 refill using Underground Argon (UAr).

The main steps required for the procedure include:

- ✓Leak test of DS-50 system.
- ✓Commissioning of custom getter.
- ✓Commissioning of the recovery system.
- ✓Draining of AAr from DS-50.
- ✓Purging of AAr from DS-50.
- ✓Filling with UAr DS-50 with UAr.

4.Plants in Use and Reference Documents

The plants in use are: the custom getter system, the cooling tower, the recovery system, and DS-50. The reference documents are the DS-50 master sheets, available at the reference source for the P&IDs, [DarkSide DocDB Document 314](#).

5.Distribution list

- ✓DarkSide Collaboration through the database [Darkside-DocDb.fnal.gov](#).

6.References

- ✓D. Lgs. 81/08 and later modifications.
- ✓D. Lgs. 334/99 and later modifications.
- ✓LNGS Safety Procedure (Sistema Gestione Sicurezza - SGS).
- ✓LNGS Enviromental Procedure (Sistema Gestione Ambientale - SGA).

7.Hazards of Unit Operations & Safety Instructions

A.Introduction

The plants of the Experiment should be operated only with management approval, and only at agreed scheduled times, with authorized personnel, and only for the specific operations planned.

Regular operation of the plants (i.e. using thermal oil/PC and/or any pumps) requires the minimum of 2 authorized persons. Authorized operators are the operators which have been trained for technical specific purpose and safety. For each shift of operations, check with the shift supervisor or operations manager, that there are no scheduled access restrictions (e.g., construction works in the car tunnel or bad weather), or planned interruptions in electrical, telephone, ventilation, nitrogen, cooling water

or fire services. All operations, in progress in each shift or daily, and notes must be recorded (date and signature) in the [Operations Logbook](#).

Check that all operating and equipment manuals and P&IDs are available underground and up to date.

Check that the Borexino/DarkSide safety manual, LNGS underground site evacuation plan, and emergency contact telephone numbers are available underground.

B. Chemical Safety

Process hydrocarbons, chemicals and utilities used, or wastes produced in the process are as follows: Hot Oil, Cooling Water, Nitrogen. A summary of the hazards, precautions and first aid to be used for each of these chemicals is listed below.

✓Exposure limits and definitions

✓**PEL**. U.S. Government OSHA Permissible Exposure Limits. PEL and TLV refers to airborne concentrations measured in the breathing zone by appropriate sampling techniques.

✓**ACGIH**. American Conference of Governmental Industrial Hygienists.

✓**TLV-TWA**. The time-weighted average concentrations for a normal 8 hour workday or 40 hour work week, to which nearly all workers may be repeatedly exposed, day after day, without adverse effect.

✓**TLV-STEL**. The short term exposure limit (TLV-STEL) is the maximum concentration to which workers can be exposed for a period of up to 15 minutes continuously without suffering from (1) irritation, (2) chronic or irreversible tissue change, or (3) narcosis of sufficient degree to increase accident proneness, impair self-rescue, or materially reduce work efficiency, provided that no more than four excursions per day are permitted, with at least 60 minutes between exposure periods, and provided that the daily TLV-TWA also is not exceeded. The STEL should be considered a maximum allowable concentration or absolute ceiling not to be exceeded at any time during the 15 minute excursion period.

✓**TLV-C**. The threshold limit value - ceiling concentration that should not be exceeded even instantaneously. For most substances, e.g., irritant gases, only one category, the TLV-Ceiling, may be relevant. For other substances, either two or three categories may be relevant depending upon their physiologic action. It is important to observe that if any one of these three TLV's is exceeded, a potential hazard from that substance is presumed to exist. The TWA-STEL should not be used as engineering design criterion or considered as an emergency control of health hazards and should not be used as fine lines between safe and dangerous concentrations.

✓Definition of Species

✓**1,2,4-TriMethylBenzene (PseudoCumene, PC, CAS: 95-63-6)**. PC is a liquid at ambient conditions. It boils at 168 °C and freezes at -44 °C. It is flammable and toxic. The flash point is ~45 °C. Its properties are detailed in the MSDS available on [DarkSide DocDB Document 301](#).

✓**TrimethylBorate (TMB, also Boron TriMethOxyde, CAS: 121-43-7).** TMB is a clear liquid at room temperature. It boils at 68 °C and freezes at -34 °C. It decomposes in contact with water in methanol an boric acid. It is flammable and burns with a green flame. Its properties are detailed in the MSDS available on [DarkSide DocDB Document 301](#).

✓**DyPhenylOxazole (PPO, CAS: 97-71-7).** is a solid powder at ambient conditions. It boils at 360 °C and melts at 70 °C. It is flammable and toxic. Its flash point is above 100 °C. Its properties are detailed in the MSDS available on [DarkSide DocDB Document 301](#).

✓**Master Scintillator Solution.** The master scintillator solution is a concentrated solution of 200 g PPO per liter of PC (or PC+TMB solution). It is a liquid at ambient conditions. It is flammable and toxic. Its flash point is approximately that of PC (or PC+TMB solution).

✓**Hot Oil.** Heat for the vacuum stripping is provided by flowing hot oil flowing through heat exchangers. The hot oil temperatures will be up to 100 C. The hot oil lines should always be insulated.

✓**Water (CAS: 7732-18-5).** Chilled water for cooling is provided through a closed loop. This water is non potable. It is not considered to be a hazard. Its properties are detailed in the MSDS available on [DarkSide DocDB Document 301](#).

✓**Nitrogen (CAS: 7727-37-9).** Nitrogen gas is used for gas stripping, internal gas blanketing, and instrument control. In hall C is not considered to be a hazard, except in confined areas for the Oxygen Deficiency Hazard (ODG). Its properties are detailed in the MSDS available on [DarkSide DocDB Document 301](#).

✓**Argon (CAS: 7440-37-1).** Argon gas is displaced from the detector during draining and from the UAr rack to the detector during filling. In Hall C argon is not considered as an hazard, except in confined areas for ODH. Its properties are detailed in the MSDS available on [DarkSide DocDB Document 301](#).

C.First Aid

In case of contact with the substances write in the last paragraph act as below:

✓If inhaled

If breathed in, move person into fresh air. If not breathing give artificial respiration Consult a physician.

✓In case of skin contact

Wash off with soap and plenty of water. Consult a physician.

✓In case of eye contact

Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

✓If swallowed

Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

D.Fire fighting Measure

✓**PPO**

✓Suitable extinguishing media

Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

✓Hazardous combustion products

Hazardous decomposition products formed under fire conditions.

Carbon oxides, nitrogen oxides (NOx) Hazardous decomposition products formed under fire conditions.

✓PseudoCumene

✓Suitable extinguishing media

For small (incipient) fires, use media such as "alcohol" foam, dry chemical, or carbon dioxide. For large fires, apply water from as far as possible. Use very large quantities (flooding) of water applied as a mist or spray; solid streams of water may be ineffective. Cool all affected containers with flooding quantities of water.

✓Further information

Use water spray to cool unopened containers.

✓TriMethylBorate

✓Suitable extinguishing media

Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

✓Special hazards arising from the substance or mixture

Carbon oxides, Borane/boron oxides

✓Further information

Use water spray to cool unopened containers.

E.PC, TMB, or Hot Oil Leaking from Pipes or Equipment

As soon as possible isolate the leaking lines or equipment, shutting down plants as necessary. If possible take corrective action to minimize the leak (tighten or tape connections, direct leaks into basins using plastic sheets and absorbent etc.). Wear PPE (helmet, safety shoes, gloves, safety glasses and flash light). Clean up according procedure (See ISO14001, response to the emergency situations).

IN CASE OF ALARM FOLLOW THE EMERGENCY ACTIONS ON PLANTS RECOMMENDED IN THIS PROCEDURE AND THE EMERGENCY PLAN OF LNGS

F.Recommended major safety equipment

✓ **Portable Cylinder Air Breathing Apparatus.** At least one self-contained breathing apparatus should be provided in each building plus five in the Control Room and one at the tank car loading area. There should be at least ten units available.

✓ **Safety Showers and Eye-Wash Fountains.** There should be a safety shower and eye-wash fountain in the following area: US SKIDS (near cleaning module). The above is a minimum requirement. Additional showers should be provided at other strategic locations.

✓ **Portable Fire Extinguishers.** Number 9 pressurized foam 50 liters fire extinguishers should be provided at strategic locations. Also useful are

large foam-type extinguishers. There should be at least two units which can be easily wheeled into use when needed.

✓ **Fire Blanket.** Although the safety showers are effective in extinguishing fire on a person, a fire blanket should be provided at the control room.

✓ **Minor Equipment.** Minor safety equipment typically on hand comprises hard hats, goggles, gloves, etc., should be available in control room. During operations the work areas (hot-oil) should be isolated with signs posted:

"VIETATO IL TRANSITO A PERSONE NON AUTORIZZATE"
"AUTHORIZED PERSONNEL ONLY"

✓ **Other Safety Equipment** absorbent cloth, containment "snakes", two vacuums liquids cleaners and a containment basin.

G.External and Environmental Impact

All the operation will be done in the confined space (Hall C). The Hall C is provided with a fixed foam fire extinguisher plant operated in manual. There are also liquid sensor and sensor to measure smokes and temperature.

The nitrogen will be vented through the exhaust plant.

The operation implies the production of liquid waste that comes from the bottom of the distillation. The waste will be stored in the Borexino waste tank and will be disposed according to the Laboratory procedure PG. 06/01.

H. ODH

ODH is mitigated by the presence of fixed and portable oxygen monitors in CRH. Use of portable oxygen monitors is mandatory for all operators entering CRH.

8. Leak Test of DS-50 System

The operation consists of checking every single connection in the DS-50 system with a helium mass spectrometer at the sensitivity level of 10^{-9} mbar \times l/sec.

9. Commissioning of Custom Getter

Upon completion of the leak check, the custom getter is commissioned by circulating AAr and verifying that the temperatures reach the design values set in the control system.

10. Commissioning of Recovery System

Upon completion of the leak check, the recovery system is commissioned. Compliance with the requires speed of recovery from DS-50 will be directly evaluated during the draining of DS-50.

11. Draining of AAr from DS-50

The general alignment of valves can be found in the pertinent DS-50 Mastersheet available at [DarkSide DocDB Document 314](#). At the beginning of the procedure, the Maintenance Manager check consistency of the positioning of each individual valve with that specified in the pertinent DS-50 Mastersheet, by checking and initializing each valve on a large printout of the pertinent DS-50 Mastersheet. Any change in the position of valves during the sub-procedure must be documented on elog.

AAr is cryopumped from DS-50 to the recovery cryostat. Weighing of the recovery cryostat before and after each removal procedure is required to keep track of the AAr inventory removed from the detector.

The recommended settings for the operation of the recovery system, DS-50, and associated safety systems are:

- ✓ Pressure set point for recovery cryostat at 13.5 psi, which should bring the pressure in DS-50 at 14.5 psi when the maximum power of 160 W is applied to the butterfly heater at the bottom of DS-50.
- ✓ Bridge from DS-50 to recovery cryostat is first level of backup for DS-50, with V-OP1 set to open at 14.5 psi and to close at 14.0 psi.
- ✓ Cooling on DS-50 is second level of backup for DS-50, and is already set at 15.6 psi.
- ✓ Backup cooling on DS-50 is third level of backup for DS-50, with V-BP2 set to open at 16.2 psi and to close at 15.9 psi.
- ✓ Vent of DS-50 is fourth level of backup for DS-50, set to open at 18.25 psi and close at 18.0 psi.
- ✓ Vent of recovery is second level of backup for recovery, set to open at 18.25 psi and close at 18.0 psi.

It is paramount to ensure the complete draining of DS-50 from any form of condensed argon. The power of the butterfly heater is either turned off or throttled towards the end of procedure to ensure the absence of condensed argon by sensing pressure differences and testing pressure rises in DS-50.

12.Purging of AAr from DS-50

The general alignment of valves can be found in the pertinent DS-50 Mastersheet available at [DarkSide DocDB Document 314](#). At the beginning of the procedure, the Maintenance Manager check consistency of the positioning of each individual valve with that specified in the pertinent DS-50 Mastersheet, by checking and initializing each valve on a large printout of the pertinent DS-50 Mastersheet. Any change in the position of valves during the sub-procedure must be documented on elog.

Upon completion of the previous step:

✓For three times, the pressure of DS-50, recovery system, and all associated volumes is lowered to 0.1 bara and then the volumes are backfilled with GAN. During the pressure lowering steps, the inlet of the pump is monitored with a RGA and gas concentrations are recorded and saved on elog.

13.Filling with UAr DS-50

The general alignment of valves can be found in the pertinent DS-50 Mastersheet available at [DarkSide DocDB Document 314](#). At the beginning of the procedure, the Maintenance Manager check consistency of the positioning of each individual valve with that specified in the pertinent DS-50 Mastersheet, by checking and initializing each valve on a large printout of the pertinent DS-50 Mastersheet. Any change in the position of valves during the sub-procedure must be documented on elog.

Upon completion of the previous step:

✓In a single instance, the pressure of DS-50, recovery system, and all associated volumes is lowered to 0.1 mbara and then the volumes are backfilled with UAr processed by the custom getter. During the pressure lowering steps, the inlet of the pump is monitored with a RGA and gas concentrations are recorded and saved on elog.

14. Operations and Shifts Requirements

Leading personnel involved in the operation and contact numbers:

Name	Cellphone	E-mail
Giuseppe Bonfini	+39-328-114-7392	giuseppe.bonfini@lngs.infn.it
Augusto Brigatti	+39-340-376-7537	augusto.brigatti@mi.infn.it
Paolo Cavalcante	+39-349-099-2651	paolo.cavalcante@gmail.com
Nicola Canci	+39-328-678-7744	nicola.canci@lngs.infn.it
Marco Carlini	+39-331-940-5680	marco.carlini@lngs.infn.it
Francesco di Eusanio	+39-348-884-2104	francesco.dieusanio@lngs.infn.it
Federico Gabriele	+39-328-169-3206	federico.gabriele@lngs.infn.it
Cristiano Galbiati	+39-338-667-9111	galbiati@Princeton.EDU
Augusto Goretti	+39-338-648-2463	agoretti@Princeton.EDU
Andrea Ianni	+39-338-936-7279	ianni@princeton.edu
Cary Kendziora	+1-224-324-7547	clk@fnal.gov
George Korga	+39-328-622-0039	george.korga@lngs.infn.it
Sergio Parmeggiano	+39-339-133-9559	sergio.parmeggiano@mi.infn.it
Yury Suvorov	+39-340-862-9743	yura.suvorov@lngs.infn.it
Hanguo Wang	+1-818-357-7484	hanguo@ucla.edu